AD-A134 121 REARRANGEMENT OF CLOSO-33-(PPH3)2-3-H-1-(R)-312-IRC2B9H10 TO CLOSO-22-(P.. (U) CALIFORNIA UNIV LOS ANGELES DEPT OF CHEMISTRY AND BIOCHEMISTR.. J A DOI ET AL. 19 MAY 83 F/G 7/3 NL

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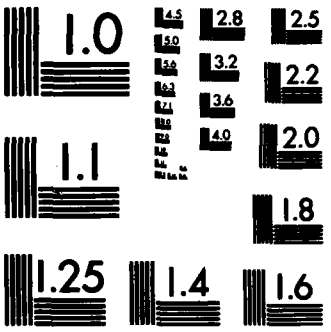

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TECHNICAL REPORT NO. 123

Rearrangement of Closo- 3,3-$(PPh_3)_2$-3-H-1-(R)-3,1,2-$IrC_2B_9H_{10}$ to Closo-2,2-$(PPh_3)_2$-H-8-(R)-2,1,8-$IrC_2B_9H_{10}$. Synthesis and X-ray Structure of Closo-2,2-$(PPh_3)_2$-2-H-8-(C_6H_5)-2,1,8-$IrC_2B_9H_{10}$.

By

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Rearrangement of *Closo*-3,3-$(PPh_3)_2$-3-H-1-(R)-3,1,2-$IrC_2B_9H_{10}$ to *Closo*-2,2-$(PPh_3)_2$-2-H-8-(R)-2,1,8-$IrC_2B_9H_{10}$. Synthesis and X-ray Structure of *Closo*-2,2-$(PPh_3)_2$-2-H-8-(C_6H_5)-2,1,8-$IrC_2B_9H_{10}$.

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During ~~our~~ the continuing investigation of the chemistry of the known alkene hydrogenation and isomerization catalyst, *closo*-3,3-$(PPh_3)_2$-3-H-3,1,2-$RhC_2B_9H_{11}$[1] (Ia), the iridium congener and its derivatives, *closo*-3,3-$(PPh_3)_2$-1-(R)-3-H-3,1,2-$IrC_2B_9H_{10}$, R = H (IIa)[1], R = C_6H_5 (IIb)[2], R = CH_3 (IIc)[2] and R = 1'-(*closo*-1',2'-$C_2B_{10}H_{11}$) (IId)[2] were prepared and notable differences between the reactivity of the Rh and Ir systems were observed. Syntheses of IIb-d, the *exo*-*nido* complexes derived from these compounds, and the dimeric complex derived from IIa will be reported in a future paper. Although thermally induced migration of carbon atoms over the surfaces of polyhedral cobaltacarboranes is a well-established phenomenon,[3] similar isomerization reactions are not so well known for other metallacarboranes. The formation of 2,1,8 isomers of icosahedral metallocarboranes have been observed previously[3] only during high temperature vapor phase thermal isomerization of (η-C_5H_5)Co($C_2B_9H_{11}$) isomers. The 2,1,8

isomer of this system was observed as the dominant kinetic product (74%) arising from the 3,1,2 isomer during such reactions at temperatures near 500° C; however, three other isomers were also detected in the reaction mixture. Here we report the ready polytopal rearrangement of IIb and IIc under mild thermolytic conditions in toluene solvent at the reflux temperature. In each of these examples the carbon atom bearing the substituent migrated in such a fashion as to produce the isomeric

closo-2,2-$(PPh_3)_2$-2-H-8-(C_6H_5)-2,1,8-$IrC_2B_9H_{10}$ (IIIb) and

closo-2,2-$(PPh_3)_2$-2-H-8-(CH_3)-2,1,8-$IrC_2B_9H_{10}$ (IIIc) species

in 84% (30 h) and 33% (9 d) yields (heating time), respectively. None of the other possible isomers were produced in detectable quantities under the experimental conditions described. The rearranged species IIId was prepared by heating $[(COD)Ir(PPh_3)_2]^+$ [nido-7-(1'-closo-1',2'-$C_2B_{10}H_{11}$)-7,8-$C_2B_9H_{11}$]$^-$ in the presence of cyclohexane at the reflux temperature (41%). The unsubstituted IIa was not observed to rearrange after 5 d at reflux in toluene. The order of relative ease of rearrangement is apparently IId>IIb>IIc in agreement with the idea that relief of steric strain provides some driving force for rearrangement by moving the bulky carbon substituent to the second belt (vertex 8) of the icosahedron, thus relieving interaction with the metal vertex and its triphenylphosphine ligands.

Below we describe the x-ray analysis of IIIb, upon which the foregoing structural arguments are based.

The structure of closo-2,2-$(PPh_3)_2$-H-8-(C_6H_5)-2,1,8-$IrC_2B_9H_{10}$ was determined by a single crystal diffraction study. The molecule is illustrated in the Figure[4]. Selected interatomic distances and angles are listed in Tables I and II. The crystal structure closely resembles that of the Rh

congener of Ia in its conformation about the metal. The most striking feature of the structure is the relative positions of the two carbon atoms of the C_2B_9 carborane ligand, which defines this complex as a member of the 2,1,8-isomer series[4]. The phenyl-bearing carbon vertex of the carborane has migrated to a position on the lower pentagonal belt, non-adjacent to the other carbon atom, resulting in a decrease in both electrostatic repulsions and steric interactions among the two carbon atoms and the Ir atom. The iridium atom, as expected, exhibits pseudo-octahedral coordination; the carborane ligand occupies three coordination sites and the two PPh_3 ligands and the hydride occupy the remaining sites. There is no fundamental distortion from the usual 12-vertex closo icosahedral geometry.

Experimental Section

All manipulations were carried out in an inert-atmosphere with standard Schlenk techniques. Unless otherwise specified all solvents were purified by standard procedures and distilled under argon before use. Baker-analyzed silica gel (60-200 mesh) was used for column chromatography with reagent grade hexane and dichloromethane solvents. Infrared spectra were recorded as Nujol mineral oil mulls on a Perkin Elmer 521 dual grating spectrometer in the region 4000- 575 cm^{-1}. ^{31}P and ^{1}H NMR spectra were obtained on a Bruker WP200 FT spectrometer. ^{11}B NMR spectra were recorded on a spectrometer built by Professor F. Anet of the Department of Chemistry, University of California, Los Angeles, CA.

Elemental analyses were by Schwartzkopf Microanalytical Laboratories, Woodside, NY. All melting points were determined on a Laboratory Devices Mel-Temp in open capillaries and are uncorrected.

Preparation of closo-2,2-$(PPh_3)_2$-2-H-8-(C_6H_5)-2,1,8-$IrC_2B_9H_{10}$ (IIIb). A 100 ml three neck flask was fitted with a reflux condenser and a gas inlet which in turn was connected to a vacuum/nitrogen manifold. The flask was stoppered, and the entire system was vacuum purged with nitrogen three times. To the flask was added 0.69 g (0.74 mmole) of IIb along with 35 ml benzene and a magnetic stir bar. The solution was heated to reflux and nitrogen gas was continually bubbled through the solution. After approximately 20 h of reflux, an additional 20 ml of toluene was added to the slurry of white solid and yellow solution. The slurry was refluxed an additional 10 h, cooled to room temperature and 0.58 g (0.62 mmole) of IIIb (84% yield) was collected. The complex was purified by column chromatography on silica gel and eluted with a 1:2 dichloromethane/hexane solvent mixture. The clear solution was reduced in volume on a rotary evaporator and yielded white microcrystals of IIIb.

Anal. Calcd for $C_{44}H_{46}B_9P_2Ir$: C, 57.07; H, 4.96; B, 10.51; P, 6.69; Ir, 20.75. Found: C, 56.90; H, 5.17; B, 10.24; P, 6.51; Ir, 20.95.

Infrared spectrum: 3060 (m), 2600 (s,sh) 2580 (vs), 2550 (vs), 2530 (s,sh), 2190 (m), 1490 (m,sh), 1480 (s), 1430 (s), 1180 (m), 1150 (m), 1085 (s), 1050 (m), 1010 (m), 990 (w), 925 (vw), 870 (vw), 815 (vw), 780 (w,sh), 760 (m), 740 (s,sh), 730 (s), 685 (vs) cm^{-1}.

NMR spectra ($): ^1H ($CDCl_3$, room temperature) -10.6, triplet $^2J_{P-H}$= 26.6 Hz (terminal Ir-H), 0.0-3.0 (terminal B-H), 7.2, multiplet (phenyl); ^{11}B (127.048 MHz, ref. $BF_3 \cdot Et_2O$, CH_2Cl_2, room temperature) -0.1, 6.3, 9.4, 20.5; $^{31}P\{^1H\}$ (10% C_6D_6 in THF, -60°C) 27.5, doublet, $^2J_{P-P}$=19.5 Hz and 25.9, doublet, $^2J_{P-P}$= 20.7 Hz.

Preparation of closo-2,2-$(PPh_3)_2$-2-H-8-(CH_3)-2,1,8-$IrC_2B_9H_{10}$ (IIIc). The complex IIc (0.31 g, 0.36 mmole) in 40 ml of toluene was treated in the same manner as described for IIb for 9 days at reflux. After 2 additional days at room temperature, white microcrystals formed and were collected, yielding 0.067 g of IIIc. The filtrate was chromatographed, eluting with 1:2 dichloromethane-hexane. The clear second band gave an additional 0.0350 g (total 0.1016 g, 0.118 mmole, 33% yield) of product.

Anal. Calcd for $C_{39}H_{44}B_9P_2Ir$: C, 54.22; H, 5.09; B, 11.26; P, 7.17; Ir, 22.25. Found: C, 53.68; H, 5.18; B, 10.30; P, 7.06; Ir, 21.76.

Infrared spectrum: 3060 (m), 2620 (w), 2560 (vs,br), 2190 (w), 1480 (s), 1430 (s), 1310 (w), 1180 (m), 1150 (m), 1085 (s), 1023 (m), 1005 (m), 995 (m), 980 (m), 930 (vw), 910 (vw), 885 (vw), 760 (m), 750 (s), 735 (s), 690 (vs) cm^{-1}.

NMR spectra (δ): ^1H ($CDCl_3$, -60°C) -10.5, triplet, $^2J_{P-H}$= 25.5 Hz (terminal Ir-H), 0.0-3.0 (terminal B-H), 1.2, singlet (methyl), 7.2, multiplet, (phenyl); ^{11}B (127.048 MHz, ref. $BF_3 \cdot Et_2O$, CH_2Cl_2, room temperature) -1.8, 7.8, 9.9, 20.7; $^{31}P\{^1H\}$ (ref. D_3PO_4, $CDCl_3$, room temperature) 12.9, singlet.

Preparation of closo-2,2-$(PPh_3)_2$-1-H-8-(1'-closo-1',2'-$C_2B_{10}H_{11}$)-2,1,8-$IrC_2B_9H_{10}$ (IIId). The salt $[CODIr(PPh_3)_2]^+$ [nido-7-(1'-closo-1',2'-$C_2B_{10}H_{11}$)-7,8-$C_2B_9H_{11}]^-$ (0.30 g, 0.46 mmole) in 35 ml of cyclohexane was heated to reflux. After 24 h the slurry became a clear red solution. After 48 h a white solid was formed. Heating was continued for an additional 4 d. The slurry was cooled to room temperature and the white solid was filtered. The product IIId (0.19 g, 41%) was collected.

Anal. Calcd for $C_{40}H_{52}B_{19}P_2Ir$: C, 48.43; H, 5.24; B, 20.70; P, 6.24; Ir,

19.37. Found: C, 48.43; H, 5.61; B, 20.14; P, 5.61; Ir, 20.00.
Infrared spectrum: 3060 (m), 2580 (vs, br), 2250 (w), 1480 (m, sh), 1475 (s, sh), 1430 (s), 1180 (m), 1150 (w), 1085 (s), 1065 (w), 1040 (w), 1020 (m), 1005 (m), 995 (m), 930 (vw), 845 (vw), 780 (w), 750 (s), 740 (s), 685 (vs) cm^{-1}.
NMR spectra (δ): ^{1}H ($CDCl_3$, room temperature) -10.7, triplet, $^{2}J_{P-H}$= 25.6 Hz (terminal Ir-H), 0.0-3.0 (terminal B-H), 7.2, multiplet (phenyl); ^{11}B (111.9 MHz, ref. $BF_3 \cdot Et_2O$, CH_2Cl_2, room temperature), 0.2, 4.5, 5.1, 11.7, 14.4, 21.4; $^{31}P\{^{1}H\}$ (ref. D_3PO_4, $CDCl_3$, room temperature) 12.0, doublet, $^{2}J_{P-P}$= 21.9 Hz and 10.7, doublet, $^{2}J_{P-P}$= 20.8 Hz.

Thermolysis of closo-3,3-$(PPh_3)_2$-3-H-3,1,2-$IrC_2B_9H_{11}$ (Ia). Ia (0.13 g, 0.15 mmole) was placed in a 25 ml 3 neck round bottom flask fitted with a water-cooled spiral condenser which was connected to a vacuum/nitrogen manifold through a gas inlet. The entire system was stoppered and vacuum purged with nitrogen three times. A magnetic stir bar and 15 ml of toluene were placed in the flask and the sample was heated to reflux. After 5 d the solution was cooled to room temperature. Only starting material was found. Constant monitoring by thin layer chromatography of the solution indicated no reaction.

Closo-2,2-$(PPh_3)_2$-2-H-8-(C_6H_5)-2,1,8-$IrC_2B_9H_{10}$ 1/2 CH_2Cl_2 was prepared as described above and crystals were grown by vapor diffusion using 1:1 chloroform/dichloromethane and hexane. A single crystal was mounted on a glass fiber. The automatic centering, autoindexing, and least-squares routines of a Syntex $P2_1$ diffractometer were used to determine the unit cell parameters of a = 11.722(4), b = 19.994(7), c = 9.923(4) Å, alpha =

100.17(3), beta = 107.59(3), gamma = 86.40(3)° and V = 2182(1) Å^3 on the basis of 15 reflections. Some details of the data collection are presented in Table III. A graphite crystal was used to provide monochromatic MoK_{alpha} radiation (0.7107 Å). The crystal density was found to be 1.48 g cm^{-3} (by flotation in CCl_4/heptane), while the calculated density was 1.47 g cm^{-3} based on Z = 2.

Intensity data (h, ±k, ± l) were collected with the theta-2theta scan technique to a limit of 2theta = 45°. Reflections were scanned at a variable rate (between 2.5 and 14.65°/min) from 1° below the MoK_{alpha1} to 1° above the MoK_{alpha2} reflection. Intensities of three standard reflections were measured after every 50 reflections. No significant deviations were observed.

The data were corrected for Lorentz and polarization effects. The intensity of a reflection I(hkl) and its estimated standard deviation sigmaI(hkl) were calculated as described previously.[5] Of 5778 unique reflections, 5025 with I>3sigma(I) were considered to be observed and were included in subsequent calculations.

Solution and Refinement of the Structure. The coordinates of the Ir atom were determined by solution of a 3-dimensional Patterson map and the structure was solved in space group $P\bar{1}$ by heavy atom techniques. The 7 phenyl groups were described as rigid C_6 hexagons with C-C = 1.391 Å and C-H = 0.998 Å. Positions for all hydrogen atoms not included as members of rigid groups were obtained from a difference map. In the final least-squares cycle 268 parameters were refined, including positional and anisotropic vibrational parameters for Ir, P, C_2B_9, and CCl_2 (at half occupancy), positional and carbon isotropic vibrational parameters for the C_6H_5 groups and positional

parameters for the remaining nongroup H atoms, with the exception of the terminal Ir-H hydride H(2). For each H atom, B was fixed at 0.5 + B of the C or B atom to which that H is attached. Atomic positional parameters for atoms not included as members of rigid groups are given in Table IV. Scattering factors and anomalous dispersion terms were taken from International Tables for X-ray Crystallography[6].

The final least-squares cycle converged at R = 0.050, Rw = 0.064 (refined by full-matrix least-squares procedure and based on F, $w = 1/\sigma^2(Fo)$; $R = \sum ||Fo| - |Fc|| / \sum |Fo|$; $Rw = [\sum w ||Fo|-|Fc||^2 / \sum w|Fo|^2]^{1/2}$; the "goodness of fit" was 2.30 as defined by $[\sum w(|Fo|-|Fc|)^2/(No-Nv)]^{1/2}$ with No = 5025 and Nv = 268. In the final cycle of least-squares refinement no shift for a nonhydrogen nonsolvate atom was larger than 0.1 of its corresponding estimated standard deviation. All calculations were performed on a UCLA Departmental DEC VAX 11/780 using the UCLA Crystallographic Package (locally edited versions of CARESS, PROFILE, ORFLS, ORFFE, and ORTEP).

Acknowledgements

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Supplementary Material Available: Listings of observed and calculated structure factor amplitudes, hydrogen positional parameters, group positional parameters and anisotropic thermal parameters of nonhydrogen atoms (pages). Ordering information is given on any current masthead page.

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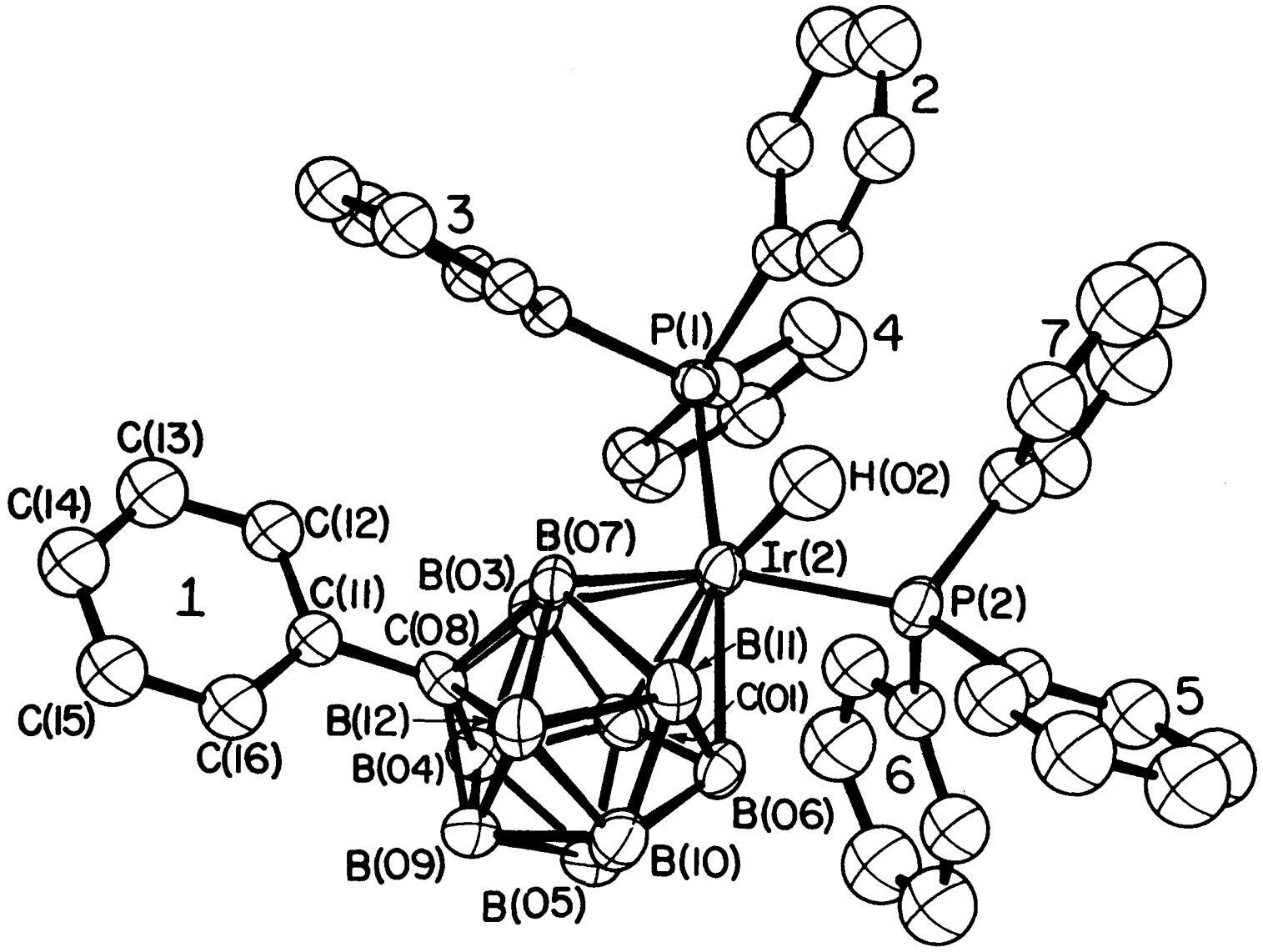
Figure Caption

The molecular structure of closo-2,2-$(PPh_3)_2$-2-H-8-(C_6H_5)-2,1,8-Ir-$C_2B_9H_{10}$ 1/2 CH_2Cl_2.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P(1)
4
7
C(13)
C(14)
C(12)
H(02)
B(07)
B(03)
Ir(2)
P(2)
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C(11)
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